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IS 1493-4 (1988): Methods of chemical analysis of iron ores, Part 4: Determination of aluminium by atomic absorption spectrophotomety [MTD 13: Ores and Raw Materials]

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(Reaffirmed 2001)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF IRON ORES

PART 4 DETERMINATION OF ALUMINIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

1. Scope — This standard (Part 4) prescribes the atomic absorption spectrophotometric method for determination of aluminium in iron ores in concentration range 0·1 to 5 percent.

2. Sampling — Sample for the determination shall be drawn and prepared in accordance with IS : 1405-1982 'Methods of sampling iron ores'

3. Quality of Reagents — Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

4. Determination of Aluminium by Atomic Absorption Spectrophotometric (AAS) Method

4.1 *Outline of the Method* — Decomposition of sample by heating with dilute hydrochloric acid. Insoluble residue hydrofluorised, fused with potassium pyrosulphate and taken up with the main solution. Aluminium is determined by AAS method.

4.2 *Preparation of Standard Aluminium Solution* — Single element standard for the preparation of the calibration curve may be used but for the sake of accuracy and to some extent eliminating the inter element effects, however, mixed standard of aluminium, iron, titanium and chromium are prepared.

4.2.1 Stock solutions of the individual elements (1 000 ppm) are prepared either from high purity salts of definite composition or from the pure metal (not less than 99·9 percent purity) by dissolving in appropriate acids, diluting the solution of a definite volume. The mixed standards of 0, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50 ppm each of iron, aluminium, titanium or chromium containing about 0·2 M tartaric acid, 2-3 N sulphuric acid, about 1 000 ppm to 10 000 ppm of potassium salts are prepared from the 1 000 ppm of standard solution of individual elements that is iron, aluminium titanium or chromium by appropriate dilutions.

4.2.1.1 *Calibration curve* — Calibration curves for aluminium (or alumina) are prepared from the above solutions and the linear portion of the curve is considered for comparision with the sample solution, maintaining nearly the same matrix.

4.2.1.2 *Blank* — Prepare a blank for the standard samples using the same quantity of reagents mentioned above (without the standard aluminium solution) and deduct the value, if any, obtained from the standard solutions.

4.3 *Preparation of Stock Solution*

4.3.1 *Reagents*:

Dilute hydrochloric acid — 1 : 1 (*v/v*).

Dilute sulphuric acid — (1 : 1), (1 : 4). (*v/v*).

Hydrofluoric acid — 40 percent.

Potassium pyrosulphate — solid.

4.3.2 *Procedure* — Weigh 0·500 g of the powdered sample (nearby to 106 µm size) in a 250 ml beaker and add about 25 ml of dilute hydrochloric acid and warm in a hot plate with the lid till the sample is decomposed. Remove the lid and add 20 ml of dilute sulphuric acid (1 : 1) and slowly evaporate to fumes. Cool, add water and warm to dissolve the salts, and filter (use close texture filter paper). Wash the residue with hot dilute sulphuric acid (1 : 4) solution and collect the filtrate and the washings in 250-ml volumetric flask. Ignite the residue in a platinum crucible and remove silica by volatilisation with hydrofluoric acid. Fuse the residue with 1 g of potassium pyrosulphate. Dissolve the melt in water and add to the filtrate in the volumetric flask. Make up the volume.

4.3.3 *Solution for AAS measurement* — Take an aliquot of the sample solution expected to contain not more than 50 ppm of alumina (10 ml in 100 ml volumetric flask). Add calculated amount of

Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee, SMDC 35 [Ref : Doc : SMDC 35 (2769)]

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sulphuric acid, tartaric acid, and potassium nitrate solution so that the final volume contain sulphuric acid (2—3 N), tartaric acid (0·2 M) and potassium salt to about 2 000 ppm. If sufficient sodium salt or potassium salt (1 000 ppm to 10 000 ppm) is present in the solution further addition is not required.

4.3.3.1 *Blank* — Prepare a blank solution, following strictly the procedure with all the reagents except the sample.

4.3.3.2 *AAS measurements* — Adjust the atomic absorption instrument using instrument parameters as recommended by the manufacturer. Use nitrous oxide acetylene flame. Aspirate water to establish that the absorbance reading is not drifting and then set the initial reading for water to zero absorbance. Aspirate blank solution, a sample solution and standard aluminium solution. When a stable response has been obtained for each solution, measure the absorbance at 309 nm. Aspirate water between each solutions. Substract the blank values from the standard as well as from the sample.

4.4 *Calculation* — Prepare a calibration curve (*see* **4.2.1.1**) by plotting net absorbance values of calibration solutions against micrograms of aluminium per millilitre. Convert the absorbance value of test solution to microgram of aluminium by means of calibration curve. Calculate the percentage of aluminium as follows:

$$\text{Aluminium, percent} = \frac{I}{W} \times 100$$

where

I = microgram of aluminium/millilitre of test solution obtained from the calibration curve, and

W = sample concentration in microgram/millilitre in the test solution.

EXPLANATORY NOTE

IS : 1493 (Part 1) - 1981 'Methods of chemical analysis of iron ores: Part 1 Determination of common constituents' covers the chemical analysis of iron ores for determination of moisture, silica, iron, phosphorus ($>$ 0·5 percent), aluminium and sulphur by conventional method, namely, gravimetric and volumetric method. The chemical analysis for determination of remaining constituents given in IS : 1493 - 1959 'Method of chemical analysis of iron ores' will be covered in Part 2 of this standard which is under preparation. With the publication of IS : 1493 (Part 2), the original IS : 1493 - 1959 will be with drawn.

Since atomic absorption spectrometry (AAS) for iron ore has greatly facilitated the determination of many of the constituents, a need was felt to publish a standard for determination of various constituents in iron ores by atomic absorption spectrometry. Determination of titanium, chromium, vanadium, calcium and magnesium in iron ores by 'Atomic absorption spectrometry' have been covered in IS : 1493 (Part 3)-1987. This standard (Part 4) covers the determination of aluminium in iron ores by AAS method. However, the determination of aluminium in iron ores by complexometric (EDTA) method has been covered in IS : 1493 (Part 1)-1981.

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